Notebook Series -- Notebooks by Edison
Notebook, N-16-11-06.1

This notebook was used by Edison during the period November-December 1916. The first entry, entitled "Dinwiddie Expt," lists twelve organic compounds with the notation "Sulfonate Calcium." Additional entries at the beginning of the book pertain to lamp black and other ingredients used in disc record manufacture and to problems with the use of lamp black in record blanks. Several groups of entries in the middle of the book relate to a series of chemical syntheses and separation experiments involving amino phenol, zinc, and "myrbane" (oil of mirbane, or nitrobenzene, used in the manufacture of anilines for textile dyeing.) Inserted into the book is a note by chemist Richard G. Berger, which contains instructions for the preparation of "brown P-Amino Phenol of Permanent Color from Black Product" and a chemical equation expressing one of the reactions involved. The front cover is labeled "Disc Record Dinwiddie Expts. 1916." The pages are unnumbered. Approximately 25 pages have been used.

Nov 6th 1916 –

Dinwoodie Experiments

No 1 Methyl Benzyl aniline
Sulphonate of Calcium

2 Monoethyl aniline
Sulfonate Calcium

3 Ortho Nitro Anisol
Sulfonate Ca

4 = Diethylaniline
Sulfonate Ca

5 Para Xylene
Sulfonate Ca

Nov 6 1916

6 Orthochloroaniline
Sulfonate Ca

7 Monoethyl-O-Chloro Toludin
Sulfonate Ca

8. Mono Ethyl-para Toludin
Sulphonate Ca

9 Monochlorohydrin
Sulfonate Ca

10 O-Phenetidin
Sulfonate Ca

11 = Anisol
Sulfonate Ca

12 = Phenetol
Sulfonate Ca

We first found Coke particles in our Lampblack 2% - Herman Lampblack [illegible] have it in - These particles Cut the soft Copper record Matrix a weaker Crackly surface found then cut by lubrication & pouring & etching. The last point was mostly grit & Lampblack people say & Calcium & are hard & then & weaker the

Nov 25/16

Coke,

We have great trouble with the Lampblack clotting on grinder which is a Raymond grinder

Have tried defloculating,

Black wont go into water without considerable beating & stirring —

LB goes into acid like H_2O distilled, easy

LB goes into alcohol also Gasolene & Kerosene easy

LB oxidized by K Bichromate hot & H_2SO_4 goes out but is still lumpy & even after washing water dries in a hard lumpy condition afterwards, had to get into alcohol —

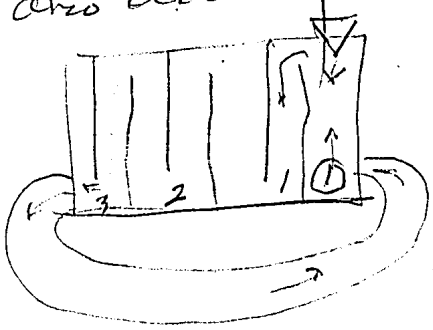
Oxidizing by KClO3 & HCl fluffs it up the first time but before the effect on Black is pronounced it defloculates it so much that its almost Coffee-Brown then fluffy & then fluffy clumps up & Alcohol scarcely get thru & then alcohol is full of single fine particles —

Doutecchio can wash out the NaCl — it dries on filter paper entirely different from the others,

On a coach glass the thin layer of LB, by transmitted light is Red brown & transparent,

We are rigging up a Wheel water cooled to burn out & make LB & scrape off —

also blowing the LB



No 2 was ok seriously as
sign of coke grit –
now using it

Made a small $2\frac{1}{2} \times 5$ high mixer
with 4 arms & springs $1\frac{1}{2}$
long reaching out & bent
round to sides & behind
& break up clots of
Lampblack in bag Vans
works fairly good = $1\frac{1}{2}$
hours only few specs noticed
Think this can be made to
work fine —

Will now try & substitute
Kieselguhr for Lampblack &
see how it covers on
blank – Coned dye with Naphthol

11/26-16 -

Today used reg blank Condensate Var
Var a Celluloid Submersion
blanks reg very looks
good, well permit + test
Dec 27/16

Melted @ heat + added
Castor Oil — It
dont blend reg
Melted Rosin + added
Castor Oil —
Blends OK
good

Rose – Paraldehyde
Vac Dried
from Chloroform

Not sol except trace in Kerosene

" Benzol

Quite sol. Acetone Chills out white, but Zinc is given burning —
May be something in it

Benzaldehyde dissolves
good — Enormous There is some Zinc
on burning but very little —
Think this is very good —

Turpentine NG

Petroleum Benzine – only a little
white —

Ether – trace —

Pyridin good solvent – nothing Chills out
VERY Red

Get some Acetic Aldehyde

If the Aldehyde HCl has scarcely any Zinc in it —

So far only practical ones are Acetone & Benzaldehyde —

Hydrochloride

Acetone dissolves it in great amounts turns red Cant chill out even a Concentrated sol
Ably Combines — Alcohol Cant chill out, turns red —
Kerosene Naphtalene Solvent Naphtha Benzol Sassafras Carbon Tetrachloride — None Dissolves any

Bisulfite Acids if Bronzes dissolved (Dont dissolve much) & then treated with HCl White Leaflets after much hard work Come out, microscopic then — used Zinc to reduce

Renzadine

~~Upright Reversible~~

~~Test~~

NO 2 Horizontal pot

Started 7.50 am 11/9/16 done at 4.50 pm used 5 hours to put Zinc in – used hot water heating at times to promote action

10 grms Magnesium
6 cc of Cobalt
25 grms Zinc dust
30 cc of 30% NaOH.

formula for both

NO 1

Started 7.35 am – 11/9/16 done 4.35 pm
5 hours put Zinc in 30 min

Benzadine -

No 1 Upright, double paddle pot

Started 7 50 am - done 4 50 pm took 5 hours to put Zinc in, 30 minutes after Zinc all in added 20 cc water took 9 hours to make the run started put at times by hot water when action got put cold

No 2 operated the same length of time - This is horizontal pot but in this case 40 cc of water was added - This pot was warm & when ~~put~~ before put water in got thick & muddy & the overflow tube stopped up - did not have this trouble with No 1 Double paddle machine

The yield of both was 54% of the theoretical - but No 1 gave a light colored 2nd Extract product whereas No 2 gave Red oil This shows

that NO 1 is best, apparently during reaction it foams terribly. I will measure the change of the empty space to see how big a pot must be used

The formula which Phal's was

10 grms Myrbane
6 cc Alcohol
25 grms Zinc dust
30 cc of a 30% NaOH sol

There is a very great amount of ZnO formed which does not dissolve in the soda & this makes it very difficult to filter. – We must proportion our NaOH to lessen it or will have trouble after filtering out. Everything this ZnO dissolves in NaOH 30% or 20% even warm –

Another trouble is that in using warm boiling alcohol to extract the alcohol gets cooled in filtering & is practically impossible to get it all out,

In working by difference of solubility the filtering apparatus must be above the boiling point of the liquid –

There is some metallic Zinc left – probably 20 @ 22 grms would have done –

If all could have been got we should probably have got 60% yield of chloralid

We only used 30 cc of 30% NaOH
whereas it requires for 25 grms
Zinc to form Zincate. 102
grams of 30% NaOH,

No 1 Upright pot Double
piccolo —
Depth - 4 inches

With 10 g magnesium
25 g Zinc
6 cc Alcohol
90 cc 30% NaOH,
40 cc water —

it made a depth of
2 3/8 leaving 1 5/8 clear
space for foaming

previous run only used
30 cc NaOH 30% & 20 cc water
This does used for measuring
had therefore 80 cc more
than 1st Run this will
show of foaming will
take place at 10/CC
also this change

Run 2

No 1 Pot,

10 grams Hydrazine
25 " " Zinc
6 cc alcohol
90 cc 30% NaOH,
40 cc water after all Zinc in.

No 2 Pot same except only 60 cc of 30% NaOH,

Use hot water both cases to keep warm as Zinc dissolves

The No 1 pot worked OK Zinc free & ZnO free small filtered fine - will have no trouble with this

No 2 more ZnO but not very much more than No 1 Did not filter as well — No 1 is preferable

Yield No 1 5.450 + 590 from Alcohol Evap. allowing for 3% bad Hydrazine 84.15% yield from Chloride.

Yield No 2 5695 + 495 from al.
x but in No 2 think little Zinc is in -

The remaining alcohol is being Evap which will give stick more -

We run 1/2 hour after last Zinc is in before putting in water —

84.15% yield from Double Cleaner pot, after came from the Horizontal

3rd Run No 1 Pat.

10 grams Myrbane
18 " Zinc
75 cc 30% NaOH
6 cc Alcohol

To be run better three (before) Zinc put in over same period 5 hours — Total 9 hours
30 cc water to be put in after all Zinc is in —

No 2 Pat same except only 15 grams Zinc —

3rd Run is OK
got 6070 white
& 493 light red
97% pure myrbane & lost slight amount in working & probably in the NaOH solution
This figures out Geller Chem 90%

The reason of high efficiency is due to fact that I kept the temp advanced 140° F over the whole time — I put in the water 1/2 hour after Zinc was all in —

We will now use for mfg

10 parts by weight Myrbane
18 " " Zinc dust
75 cc of 30% NaOH
6 cc Alcohol

9 Hour run — 5 hours to put Zinc in
5½ @ 6 hours before 30 cc water put in — Keep low temperature 140° F for the night run

Amidophenol

The Red solution from acting on base by HCl & reducing by Tin as far as possible is slide Red but light –

Shaking in separating funnel with
Gasolene NG
Benzol NG
Carbon Tetrach NG
Amyl Alcohol takes it out & Amyl " becomes deeply Colored Believe most can be got out this way – it leaves it light yellow, which may be something else –

Chloroform NG –

Benzaldehyde – NG – acts on it

Acetate Amyl separates but not near as good as amyl Al

Turpentine NG

Phenol – good –

Paranitro Xylol works pretty good & like amyl –

Dichloroethylene NG

Ortho Nitroanisol NG acts on it

Methyl benzanilin NG acts on it

Hypnon – Better than Amyl –

Oil Sassafras NG

Camphor Oil NG

Isobutyl Xylol NG

Eucalyptus shows but dont clean

Oil Lavender

Ketone Solvent No 2 dissolves out black but Colors oil probably impurs investigate – Redistill –

Ketone Solvent No 2 from Newark
Mass is darkened by HCl dilute
I have shaken it with some water
& Come HCl & separated it by
funnel so as to distill it
I distilled some get white
but no use the HCl darkens it
MG

Phenol [illegible] better than
Hypnon but different results
odet phenol — Tried Zangof
to and it [illegible] do it —

If shaken with some black
first & [illegible] & then
shaken with Hypnon it
becomes almost white
but not more than phenol-bayol
Externally Z [illegible]

Big Tank —

for every $\frac{1}{4}$ inch 500 cc —

12 inches gives 24000,

100 cc Myrbane weighs
120 grams

3 grms Zinc dust to 1 cc

10 grms Myrbane	8.33 cc
75 cc 30% Soda	75 cc
6 cc Alcohol	6 cc
18 grms Zinc dust	6 cc
30 cc water	30 cc

1 unit 125.33 cc

Tank multiply 192 times

Fortlies Pat.

1600 cc Myrbane
1152 cc Alcohol
14400 cc 30% Soda
3456 grms Zinc dust,

Put 173 grams of Zinc in every 15 minutes, this will take 5 hours,

After all Zinc in add 5760 cc water & run 4 hours more

Altogether 9 hours —

Works OK, now red stuff —

Pat & proportions apparently OK - am testing for yield

Will plate Zinc out

On account of 21% attaching the paper on filter when cloth we used 1/16 sheet asbestos putting cloth over halves of 6" filter & then asbestos sheet. This works OK — asbestos has too short & weak fibre to stand vac but cloth holds it OK

We filter out 95% of the strong solution washed out & dilute it =
Then solution was water plated out the Zinc on 300 mandrels by high density & slow moving wheel cathode & scraper
This works OK on small wheel —

[ITEMS(S) FOUND IN BOOK]

Nov. 23, 1916.

Preparation of brown p-Amino Phenol of Permanent Color from Black Product.

Dissolve black p-Amido-phenol in conc HCl and boil. Decant of the clear solution and obtain the crude hydrochloride by chilling in ice-water. Filter, sucking the product dry as possible.

Dissolve the crude greyish black hydrochloride in a minimum of cold water. Filter: Add to the clear solution a saturated solution of sodium sulfite which has been made slightly acid towards phenolphthalein. This precipitates brown p-amino-phenol. Filter on a suction funnel and finally wash once or twice with a minimum of ice cold water. This final washing removes excess of sodium sulfite.

Laboratory of Thos. A. Edison

[ITEMS(S) FOUND IN BOOK]

Reaction for the action of sodium sulfite upon the hydrochloride is represented by the following:

$$C_6H_4\begin{matrix}-NH_2 \cdot HCl \\ -OH\end{matrix} + Na_2SO_3 \rightarrow C_6H_4\begin{matrix}-NH_2 \\ -OH\end{matrix} + NaCl + NaHSO_3$$

p-amido-phenol hydrochloride

p-amido-phenol